phorus ligands, as well as investigating the mechanism of these unusual coupling reactions. The results of these studies will be reported in due course.

**Acknowledgment.** Thanks are expressed to the donors of the Petroleum Research Fund, administered by the American Chemical Society, and Monsanto Co. for support of this work. The author acknowledges receipt of a Missouri Research Assistance Act grant and the award of a Faculty Research Fellowship. Funds from the National Science Foundation to the University of Missouri-St. Louis for the purchase of a NMR spectrometer are gratefully acknowledged. Thanks are also expressed to Johnson Matthey for a generous loan of platinum salts, to Dr. R. J. Cross for helpful discussions, and to NATO for a research grant.

# Preparation of Vinylstannanes via the Peterson Reaction

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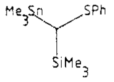
The condensation of anions derived from (phenylthio)(tri-*n*-butylstannyl)(trimethylsilyl)methane (**2a**), *tert*-butyl (tri-*n*-butylstannyl)(trimethylsilyl)acetate (**2b**), and (tri-*n*-butylstannyl)(trimethylsilyl)methane (**2c**) with carbonyl compounds was used to compare the Peterson and tin eliminations. In all cases, the silicon moiety was eliminated to give the vinylstannane **4**.

A recent paper,[1] describing the elimination of silicon from a $\beta$-silyl-$\beta$-stannylalkoxide (1) derived from condensation of an $\alpha$-silyl-$\alpha$-stannyl ester enolate with a carbonyl compound, prompts us to report our results in this area.

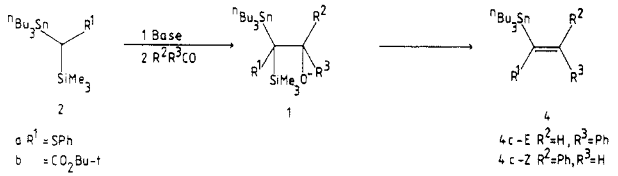
The Peterson reaction[2] has found acceptance as a useful alternative to the Wittig reaction. By contrast, the tin analogue of this elimination has found little use in synthesis although it is known[3] and has been shown to exhibit stereochemical control.[4] Previous studies suggested that the route outlined in Scheme I could provide a general method to vinylstannanes.[1,5]

Our initial studies were carried out with the sulfide **2a** which is readily available from (phenylthio)(trimethylsilyl)methane (**3**).[6] Anion formation with potassium diisopropylamide (KDA), or lithium diisopropylamide (LDA) in the presence of hexamethylphosphoric triamide (HMPA), followed by condensation with a nonenolizable carbonyl compound gave the vinylstannanes (**4a**, $R^2$ = Ph, $R^3$ = H, 63%; $R^2$ = $R^3$ = Ph, 27%). No stereoselectivity was seen, nor was any vinylsilane detected (NMR, GLC, TLC); the balance of material was **2a** together with a small amount (ca. 10%) of **3**. Enolizable carbonyl compounds were deprotonated by the anion which is also a base. Some aldehydes (e.g., hexanal), however, did give low yields of **4a** (<20%); the amount of destannylation increased in these cases. The trimethylstannyl compound **5** has been shown to react in an analogous manner, although the yields were higher with enolizable aldehydes.[5]



5

### Scheme I



The $\alpha$-silyl-$\alpha$-stannyl ester **2b** was prepared by a similar method to that already described[7] from *tert*-butyl (trimethylsilyl)acetate.[1] The tin moiety had to be introduced last to circumvent the problem of O-silylation. The $\alpha$-stannyl esters were relatively difficult to purify; significant destannylation occurred under mild conditions, such as chromatography on silica. Again, the anion was prepared by treatment with KDA or LDA-HMPA and gave good yields with nonenolizable carbonyl compounds (**4a**, $R^2$ = Ph, $R^3$ = H, 72%; $R^2$ = $R^3$ = Ph, 46%) but tended to act as a base with enolizable aldehydes and ketones.

Finally, the unsubstituted derivative (tri-*n*-butylstannyl)(trimethylsilyl)methane (**2c**) was prepared by condensation of ((trimethylsilyl)methyl)magnesium chloride with tri-*n*-butyltin chloride in 95% yield; this procedure has the advantage of cheaper reagents than the previously reported route.[8] Deprotonation of **2c** proved troublesome; not a surprising observation as bis(trimethylsilyl)methane is also difficult to deprotonate,[5] and use of alkyllithiums result in transmetalation of a stannyl group.[9] No reaction occurred between **1c** and LDA, but deprotonation was achieved with KDA—albeit in 45% yield as detected by deuteration; some destannylation (ca. 20%) also occurred. Condensation of this anion derived from **1c** with benzaldehyde led to the vinylstannane (**4c**, $R^2$, $R^3$ = H, Ph; 35%; an *E*:*Z* ratio of 1:1 by NMR). No vinylsilane was detected. Reactions with enolizable carbonyl compounds resulted in deprotonation of the latter.

Small amounts of destannylated alkenes were detected from the reactions of **2a** and **2c**. This observation suggests

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[1] Zapata, A.; Fortoul, R. C.; Acuna, A. C. *Synth. Commun.* **1985**, *15*, 179.

[2] For a review see: Ager, D. J. *Synthesis* **1984**, 384.

[3] Murayama, E.; Kikuchi, T.; Sasaki, K.; Sootome, N.; Sato, T. *Chem. Lett.* **1984**, 1897. Sato, T.; Kikuchi, T.; Sootome, N.; Murayama, E. *Tetrahedron Lett.* **1985**, *26*, 2205.

[4] Kauffmann, T.; Kriegsmann, R.; Altepeter, B.; Steinseifer, F. *Chem. Ber.* **1982**, *115*, 1810. Davis, D. D.; Gray, C. E. *J. Org. Chem.* **1970**, *35*, 1303.

[5] Grobel, B. T.; Seebach, D. *Chem. Ber.* **1977**, *110*, 852.

[6] Ager, D. J. *Tetrahedron Lett.* **1981**, *22*, 2803. Ager, D. J. *J. Chem. Soc., Perkin Trans. 1*, in press.

[7] Zapata, A.; Acuna, A. C. *Synth. Commun.* **1984**, *14*, 27.

[8] Seitz, D. E.; Zapata, A. *Tetrahedron Lett.* **1980**, *21*, 3451.

[9] Ager, D. J. *J. Chem. Soc., Perkin Trans. 1* **1983**, 1131.

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that the destannylated α,β-unsaturated esters observed from **2b**[1] arise from destannylation of **2b**, and subsequent condensation of the α-silyl ester enolate with the carbonyl compound, rather than destannylation of the intermediate **1b**.

## Experimental Section

All reactions involving organometallic reagents were carried out under an atmosphere of dry nitrogen. Tetrahydrofuran (THF) and diethyl ether were distilled from sodium benzophenone just prior to use. NMR spectra (60, 90, and 220 MHz) were recorded in deuteriochloroform solution with dichloromethane as internal standard.

Petroleum ether refers to that fraction with a boiling point of 60–90 °C.

**Preparation of (Phenylthio)(tri-*n*-butylstannyl)(trimethylsilyl)methane (2a).** Butyllithium (36 mL of a 1.4 M solution in hexane, 50 mmol) was added to (phenylthio)(trimethylsilyl)methane[9] (**3**) (9.8 g, 50 mmol) in THF (100 mL) at 0 °C. After the solution had been stirred for 0.5 h, tri-*n*-butyltin chloride (14.9 mL, 17.9 g, 55 mmol) was added. The mixture warmed to room temperature overnight, poured into saturated aqueous ammonium chloride (350 mL), washed with saturated aqueous sodium chloride (150 mL), dried ($Na_2SO_4$), and evaporated under reduced pressure to give, after column chromatography ($SiO_2$; 500 g; 1100 × 30 mm, eluting with petroleum ether) the stannane **2a** as an oil (20.6 g, 85%): $R_f$ (petroleum ether) 0.4; IR $\nu_{max}$ ($CHCl_3$) 1590 (ArC=C), 1250 cm$^{-1}$ ($SiMe_3$); NMR δ 7.5–7.0 (5 H, m, Ph), 2.10 (1 H, s, CHSSiSn), 1.90–0.7 (27 H, m, $SnBu_3$), 0.15 (9 H, s, $SiMe_3$); MS, *m/z* (relative intensity) 484 (6, $M^+$), 73 (100, $Me_3Si^+$). Anal. Calcd for $C_{22}H_{42}SSiSn$: C, 54.55; H, 8.55. Found: C, 54.8; H, 8.7.

**Reaction of the Anion Derived from 2a with Carbonyl Compounds.** *n*-Butyllithium (7 mL of a 1.4 M solution in hexane, 10 mmol) was added to diisopropylamine (1.40 mL, 1.01 g, 10 mmol) and potassium *tert*-butoxide (1.12 g, 10 mmol) in THF (25 mL) at −78 °C.[10] After 5 min, the stannane **2a** (4.84 g, 10 mmol) in THF (5 mL) was added and, after a further 0.25 h at this temperature, the carbonyl compound (10 mmol). The mixture warmed to room temperature overnight. The reaction was quenched by pouring into saturated aqueous ammonium chloride (50 mL). The mixture was extracted with ether (3 × 50 mL), and the combined extracts were washed with saturated aqueous sodium chloride (30 mL), dried ($Na_2SO_4$), and evaporated under reduced pressure to give, after flash chromatography (eluting with petroleum ether–$CH_2Cl_2$), the vinylstannanes **4**. Benzaldehyde gave 1-(tri-*n*-butylstannyl)-1-(phenylthio)-2-phenylethene (**4a**, $R^1$ = SPh, $R^2$ = Ph, $R^3$ = H) as an oil (3.06 g, 63%): $R_f$ ($CH_2Cl_2$) 0.4; IR $\nu_{max}$ ($CHCl_3$) 1590 cm$^{-1}$ (ArC=C); NMR δ 7.6–7.0 (ca. 5.5 H, m, Ph and (*Z*)-CH), 6.81 (ca. 0.5 H, (*E*)-CH), 1.9–0.6 (27 H, m, $SnBu_3$); MS, *m/z* (relative intensity) 500 (2, $M^+$), 110 (100, PhSH). Anal. Calcd for $C_{26}H_{38}SSn$: C, 62.3; H, 7.65. Found: C, 62.0; H, 7.5. Benzophenone yielded 2,2-diphenyl-1-(tri-*n*-butylstannyl)-1-(phenylthio)ethene (**4a**, $R^1$ = SPh, $R^2$ = $R^3$ = Ph) (1.56 g, 27%) as rhomboids: mp 68–70 °C (petroleum ether); $R_f$ ($CH_2Cl_2$) 0.4; IR $\nu_{max}$ ($CHCl_3$) 1590 cm$^{-1}$ (ArC=C); NMR δ 7.8–7.0 (15 H, m, Ph's), 1.9–0.7 (27 H, m, $SnBu_3$); MS, (relative intensity) *m/z* 576 (1, $M^+$), 110 (100, PhSH). Anal. Calcd for $C_{32}H_{42}SSn$: C, 66.55; H, 7.35. Found: C, 66.2; H, 7.1. The condensation could also be accomplished by deprotonation of the stannane **2a** (10 mmol) with lithium diisopropylamide (10 mmol) in THF (25 mL) and HMPA (3 mL) at −78 °C for 0.5 h. The yields were, with benzaldehyde, 51%, and benzophenone, 16%, respectively.

**Preparation of *tert*-Butyl (Trimethylsilyl)(tri-*n*-butylstannyl)acetate (2b). Method A.**[1,9] *tert*-Butyl (trimethylsilyl)acetate[11] (4.7 g, 25 mmol) was added to a solution of lithium diisopropylamide (25 mmol prepared from diisopropylamine, 3.5 mL, 2.5 g, 25 mmol, and *n*-butyllithium, 17.9 mL of a 1.4 M solution in hexane) in THF (50 mL) at −78 °C. After 0.5 h, tri-*n*-butyltin chloride (6.8 mL, 8.15 g, 25 mmol) was added and the mixture allowed to warm to room temperature overnight. The reaction mixture was poured into saturated aqueous ammonium chloride (50 mL) and ether (50 mL). The organic layer was separated while the aqueous layer was extracted with ether (2 × 50 mL). The combined extracts were washed with saturated aqueous sodium chloride (30 mL), dried ($Na_2SO_4$), and evaporated under reduced pressure to give the ester **2b** (11.3 g, ca. 90% pure by NMR). All attempts to purify this compound by distillation or chromatography resulted in degradation with the accompanying loss of purity. The ester was used, therefore, without further purification: IR $\nu_{max}$ ($CHCl_3$) 1695 cm$^{-1}$ (C=O); NMR δ 1.8–0.6 (37 H, m overlaid with singlets at δ 1.54, CHSiSn, and δ 1.34, *t*-Bu) and 0.10 (9 H, s, $SiMe_3$).

**Method B.** *tert*-Butyl acetate (3.37 mL, 2.9 g, 25 mmol) was added to a solution of lithium diisopropylamide (25 mmol prepared from diisopropylamine, 3.5 mL, 2.5 g, 25 mmol, and *n*-butyllithium, 17.9 mL of a 1.4 M solution in hexane) in THF (50 mL) at −78 °C. After 0.5 h, chlorotrimethylsilane (3.0 mL, 2.6 g, 24 mmol) was added and the mixture stirred for 1 h at −78 °C. After this time, a solution of lithium diisopropylamide (25 mmol) in THF (10 mL) was added and, after a further 1 h at −78 °C, tri-*n*-butyltin chloride (5.4 mL, 6.5 g, 20 mmol). The reaction mixture was allowed to warm to room temperature overnight and poured into saturated aqueous ammonium chloride (50 mL) overlaid with ether (50 mL). The organic layer was separated while the aqueous layer was extracted with ether (2 × 50 mL). The combined extracts were washed with saturated aqueous sodium chloride (30 mL), dried ($Na_2SO_4$), and evaporated under reduced pressure (50 °C (0.5 mmHg)) to give **2b** (8.2 g, ca. 80% pure by NMR) which was used without further purification.

**Reaction of the Anion Derived from 2b with Carbonyl Compounds.** The condensations were carried out as described for the sulfide **2a**. Benzaldehyde gave *tert*-butyl 3-phenyl-2-(tributylstannyl)propanate (**4b**, $R^1$ = $CO_2$-*t*-Bu, $R^2$ = Ph, $R^3$ = H)[1] (3.19 g, 72% based on purity of **2b** noted above): IR $\nu_{max}$ ($CHCl_3$) 1690 cm$^{-1}$ (C=O); NMR δ 8.30 (ca. 0.5 H, s, CH=), 7.3 (5 H, br s, Ph), 6.61 (ca. 0.5 H, s, CH=), 1.50 and 1.41 (9 H, 2s, *t*-Bu) overlaid on δ 1.8–0.7 (27 H, m, $SnBu_3$). Benzophenone yielded *t*-butyl 3,3-diphenyl-2-(tributylstannyl)propenate (**4b**, $R^1$ = $CO_2$-*t*-Bu, $R^2$ = $R^3$ = Ph) (2.35 g, 46%) as an oil, which in our hands would not crystallize: $R_f$ ($CH_2Cl_2$) 0.4; IR $\nu_{max}$ ($CHCl_3$) 1690 cm$^{-1}$ (C=O); NMR δ 7.7–7.0 (10 H, m, Ph's), 1.52 (9 H, s, *t*-Bu) overlaid on 1.9–0.7 (27 H, m, $SnBu_3$); MS, *m/z*, (relative intensity) no $M^+$, 57 (100, *t*-$Bu^+$). Anal. Calcd for $C_{31}H_{46}O_2Sn$: C, 65.4; H, 8.15. Found: C, 65.1; H, 8.0.

**Preparation of (Tri-*n*-butylstannyl)(trimethylsilyl)methane (2c).** (Trimethylsilyl)methyl chloride (5.7 mL, 5 g, 41 mmol) was added to magnesium turnings (1.0 g, 42 mmol) and a crystal of iodine in ether (30 mL) to form the Grignard reagent. After the solution was heated under reflux for 1 h, tri-*n*-butyltin chloride (13.3 g, 11.2 mL, 41 mmol) in ether (5 mL) was added and the mixture heated under reflux for 18 h. Saturated aqueous ammonium chloride (25 mL) was added and the organic layer separated. The aqueous layer was extracted with ether (2 × 25 mL). The combined extracts were washed with saturated aqueous sodium chloride (30 mL), dried ($Na_2SO_4$), and evaporated under reduced pressure to give, after distillation, the stannane **2c**[10] (14.7 g, 95%): bp 164–170 °C (10 mmHg); IR $\nu_{max}$ (thin film) 1250 cm$^{-1}$ ($SiMe_3$); NMR δ 1.8–0.7 (27 H, m, $SnBu_3$), 0.08 (9 H, s, $SiMe_3$), −0.36 (2 H, s, $CH_2$).

**Anion Formation with 2c.** *n*-Butyllithium (7 mL of a 1.4 M solution in hexane, 10 mmol) was added to diisopropylamine (1.40 mL, 1.01 g, 10 mmol) and potassium *tert*-butoxide (1.12 g, 10 mmol) in THF (25 mL) at −78 °C.[10] After 5 min, the stannane **2c** (3.77 g, 10 mmol) in THF (5 mL) was added and, after a further 0.25 h at this temperature, deuterium oxide (0.27 mL, 0.3 g, 15 mmol). The mixture, warmed to room temperature overnight, was poured into saturated aqueous ammonium chloride (50 mL) and extracted with ether (3 × 50 mL). The combined extracts were washed with saturated aqueous sodium chloride (30 mL), dried ($Na_2SO_4$), and evaporated under reduced pressure, to give after flash chromatography (petroleum ether) the stannane **2c** (60% deuterated as judged by the integral of the $CH_2$ peak, of 2.83 g, 75%; 45% deuteration overall); $R_f$ (petroleum ether) 0.7.

**Reaction of the Anion Derived from 2c with Carbonyl Compounds.** The anion was generated as described above and treated with the carbonyl compound instead of deuterium oxide.

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[10] c.f. Raucher, S.; Koolpe, G. A. *J. Org. Chem.* **1978**, *4*, 3794.

[11] Hartzell, S. L.; Sullivan, D. F.; Rathke, M. W. *Tetrahedron Lett.* **1974**, 1403.

The vinylstannanes **4c** were purified by flash chromatography (petroleum ether). Benzaldehyde gave 2-(tri-*n*-butylstannyl)-1-phenylethene (**4c**, $R^2$, $R^3$ = H, Ph) (1.37 g, 35%) as a mixture of the *E*- and *Z* isomers (ca. 55:45 by NMR):[12] $R_f$ (petroleum ether) 0.4; IR $\nu_{max}$ ($CHCl_3$) 1590 $cm^{-1}$ (ArC=C); NMR $\delta$ 7.61 (ca. 0.5 H, d, *J* = 13 Hz (*Z*)-C*H*Ph), 7.5–7.1 (5 H, m, Ph), 6.87 (ca. 1 H, br s, (*E*)-C*H*=C*H*), 6.12 (ca. 0.5 H, d, *J* = 13 Hz (*Z*)-CHSn), 1.7–0.7 (27 H, m, $SnBu_3$).

**Acknowledgment.** The financial support of the Research Corp. and a University of Toledo FRAF award are gratefully acknowledged.

**Registry No.** **2a**, 79409-19-1; **2b**, 98830-88-7; **2c**, 77425-85-5; **4a** ($R^1$ = SPh, $R^2$ = Ph, $R^3$ = H), 103533-68-2; **4a** ($R^1$ = SPh, $R^2$ = $R^3$ = Ph), 103533-69-3; **4b** ($R^1$ = $CO_2$-*t*-Bu, $R^2$ = Ph, $R^3$ = H), 98830-92-3; **4b** ($R^1$ = $CO_2$-*t*-Bu, $R^2$ = $R^3$ = Ph), 103533-70-6; **4c** ($R^1$ = $R^2$= H, $R^3$ = Ph), 66680-88-4; **4c** ($R^1$ = $R^3$ = H, $R^2$ = Ph), 66680-87-3; tri-*n*-butyltin chloride, 1461-22-9; (phenylthio)(trimethylsilyl)methane, 17873-08-4; *tert*-butyl (trimethylsilyl)acetate, 41108-81-0; (trimethylsilyl)methyl chloride, 2344-80-1; *tert*-butyl acetate, 540-88-5; chlorotrimethylsilane, 75-77-4; benzaldehyde, 100-52-7; benzophenone, 119-61-9.

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[12] Leusink, A. J.; Budding, H. A.; Marsma, J. W. *J. Organomet. Chem.* **1967**, *9*, 285. Hibino, J. I.; Matsubara, S.; Morizawa, Y.; Oshima, K.; Nozaki, H. *Tetrahedron Lett.* **1984**, *25*, 2151. Labadie, J. W.; Stille, J. K. *J. Am. Chem. Soc.* **1983**, *105*, 6129.

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# *Communications*

## Synthesis of $(C_5H_5)Fe(CO)_2(\eta^1\text{-}C_5H_7)$, $(C_5H_5)Fe(CO)(\eta^3\text{-}C_5H_7)$, and $(C_5H_5)Fe(\eta^5\text{-}C_5H_7)$ Involving the $\eta^1 \rightarrow$ Syn-$\eta^3 \rightarrow \eta^5$ Route

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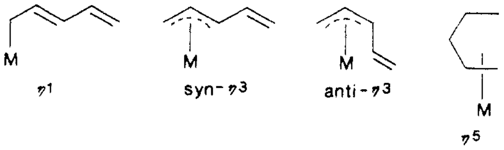
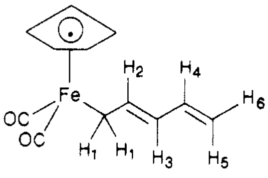
*Received November 26, 1985*

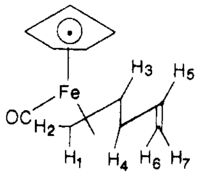
*Summary:* Reaction of $(C_5H_5)Fe(CO)_2Na$ with *trans*-1-chloropenta-2,4-diene in tetrahydrofuran at −78 °C gives $(C_5H_5)Fe(CO)_2(\eta^1$-2,4-pentadienyl) (**1**) in good yields. Photolysis of **1** in ether at −20 °C yields a mixture of $(C_5H_5)Fe(CO)(\eta^3$-pentadienyl) (**2**) and $(C_5H_5)Fe(\eta^5$-pentadienyl) (**3**). Compound **2** exists as two stereoisomers characterized as endo syn-$\eta^3$ and exo syn-$\eta^3$ by its $^1H$ NMR and IR spectra. The endo form undergoes facile isomerism to exo form at ambient temperatures. The conversion of **2** to **3** is effected by photolysis. A half-open-sandwich structure is assigned to **3** as inferred from its $^1H$ and $^{13}C$ NMR spectra.

In recent years, there is growing interest in the chemistry of acyclic pentadienylmetal complexes.[1] One of the intriguing features of these complexes is a variety of geometries for the pentadienyl group bound to a metal center as represented below. Although many recent papers[2] have described the structures of transition-metal pentadienyl compounds of the syn-$\eta^3$, anti-$\eta^3$, and $\eta^5$ configurations, the transition-metal $\eta^1$-pentadienyl compounds are rare.[3] In this communication, in addition to the report on the iron $\eta^1$-pentadienyl complex Fp–($\eta^1$-2,4-pentadienyl) (Fp = $(C_5H_5)Fe(CO)_2$ (**1**), we also describe the conversion of **1** to $Fe(C_5H_5)(CO)$(*syn*-$\eta^3$-pentadienyl) (**2**) and further to $Fe(C_5H_5)(\eta^5$-pentadienyl) (**3**).

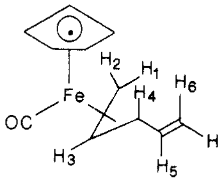




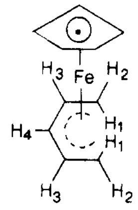
1



2 (exo isomer)



2 (endo isomer)



3

When a tetrahydrofuran solution of *trans*-1-chloropenta-2,4-diene was stirred with $(C_5H_5)Fe(CO)_2Na$[4] at −78 °C for 3 h, an air-sensitive, orange oil of 1 was obtained in higher yields after workup.[5] Analytical and spectroscopic data[6] indicate the structure of 1 as shown.

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[1] For recent reviews of acyclic pentadienyl metal complexes see: (a) Ernst, R. D. *Acc. Chem. Res.* **1985**, *18*, 56. (b) Yasuda, H.; Nakamura, A. *J. Organomet. Chem.* **1985**, *285*, 15.

[2] Bleeke, J. R.; Kotyk, J. J. *Organometallics* **1985**, *4*, 194. and references therein.

[3] Only one compound is known $Cp_2Zr(\eta^1$-2,4-pentadienyl$)_2$: see Yasuda, H.; Nagasuna, K.; Asami, K.; Nakamura, A. *Chem. Lett.* **1983**, 955.

[4] $(C_5H_5)Fe(CO)_2Na$ was prepared by reduction of $(C_5H_5)_2Fe_2(CO)_4$ (1.5 g, 4.2 mmol) with sodium amalgam (55 g, 5% sodium contents) in 50 mL of tetrahydrofuran.

[5] In a typical preparation, *trans*-1-chloropenta-2,4-diene (1.2 g, 11.6 mmol) was added dropwise to 50 mL of tetrahydrofuran solution containing $(C_5H_5)_2Fe(CO)_2Na$ (8.4 mmol) at −78 °C and stirred for 3 h. After the insoluble sodium amalgam was filtered out at −78 °C, the solution was warmed to 0 °C and evaporated to dryness to yield dark green residues. The residues were extracted with 20 mL of pentane three times, filtered, and evaporated to dryness. Using pentane as eluting solvent, the residues were chromatographed through neutral alumina column (60 g, Merck) at 23 °C. In addition to an immobile band of $(C_5H_5)_2Fe_2(CO)_4$, a gold-yellow band was eluted rapidly and collected. After the solution was removed, followed by vacuum distillation ($7.0 \times 10^{-3}$ torr, 28 °C) of the residue, an orange oil of 1 (1.39 g, 5.70 mmol) was obtained.

[6] Anal. Calcd for $C_{12}H_{12}FeO_2$: C, 59.05; H, 4.95. Found: C. 59.14; H, 5.07. Mass spectrum (12 eV): *m/e* 244 ($M^+$), 216 $(M - CO)^+$, 188 $(M - 2CO)^+$, 177 $(M - C_5H_7)^+$, 149 $(M - C_5H_7 - CO)^+$, 121 $(M - C_5H_7 - 2CO)^+$. IR (pentane): $\nu$(CO) 2010 (s) and 1956 (s) $cm^{-1}$; $\nu$(C=C) 1625 (w) $cm^{-1}$. $^1H$ NMR (100 MHz, $C_6D_6$): $\delta$ 2.16 (2 H, d, $H_1$, $J_{12}$ = 6 Hz), 3.95 (5 H, s, $C_5H_5$), 5.05 (1 H, dd, $H_6$, $J_{46}$ = 11 Hz, $J_{56}$ = 1 Hz), 5.20 (1 H, dd, $H_5$, $J_{45}$ = 16 Hz, $J_{56}$ = 1 Hz), 5.60–6.70 (3 H, complex m, $H_2$, $H_3$, and $H_4$). $^{13}C$ NMR ($C_6D_6$, 25.14 MHz): $\delta$32.4 ($CH_1H_1$), 68.2 ($CH_2$), 79.6 ($C_5H_5$), 115.1 ($CH_5H_6$), 124.7, 146.63 ($CH_3$ and $CH_4$), and 217.3 (CO).

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